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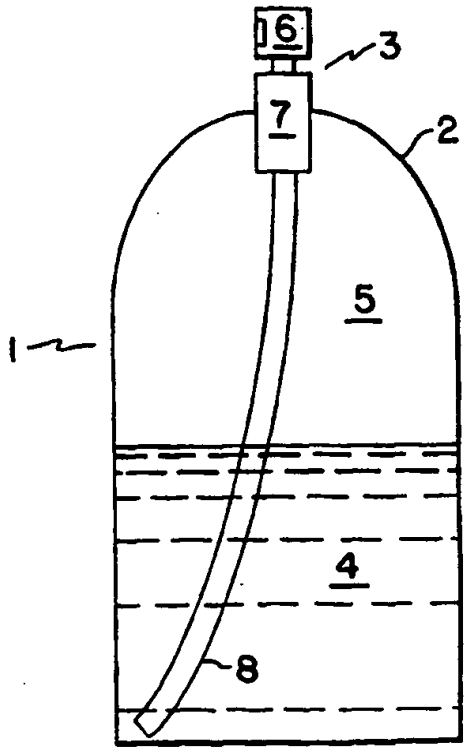
(54) Title: DISPENSING DISPERSED POWDER WHICH RESOLUBILIZES ON APPLICATION

(57) Abstract

The dispensing system (1) includes a container (2). Container (2) is a sealed enclosure to which is fitted externally actuatable dispensing means (3) for dispensing product when desired from within container (2) to the ambient atmosphere or to a target surface. Within container (2) are a dispersion (4) of active solids in a homogeneous mixture of solvent and propellant (5). Dispensing means (3) includes a button (6), valve means (7), and dip tube (8). Valve means (7) is preferably of the type having a passage for product being dispensed, and a valve operatively coupled to a spring means which urges the valve to a normally closed position preventing passage of product therethrough except when the valve is urged to an opening position by external actuation.



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DISPENSING DISPERSED POWDER WHICH RESOLUBILIZES ON APPLICATION

The present invention relates to dispensing systems. It relates more particularly to dispensing systems in which a sealed container contains a product of an insoluble active ingredient and solvent therefor all mixed with a volatile propellant or liquid. In a suitable dispensing system, the product is released, typically in the form of a stream, mist, foam, or fog of droplets having active ingredient redissolved in the solvent.

Dispensers of this type have often been employed for dispensing liquid solutions. Typically, the container holds the solution under pressure exerted by the propellant. When the solution is dispensed from the container upon actuation of the valve means, it remains in solution as it emerges from the container and is applied to a target surface as a solution. In addition, pressurized dispensing systems have also been used for dispensing finely divided solids. The solids are held within the container dispersed in liquefied propellant and/or in a liquid carrier. When the dispersion is dispensed from the container upon actuation of the valve means, the solids emerge for application to a target surface. Examples of products which are dispensed in this manner include antiperspirants, paints and other surface treatments, lubricants, pesticides, and so forth.

The prior art relating to aerosol dispensing of antiperspirants illustrates the manner in which pressurized dispensers have heretofore been used to dispense solutions and dispersions of solids.

Antiperspirants have classically presented a challenge to dispense because they can be difficult to solubilize.

Thus, the approaches heretofore taken to formulating systems for dispensing active ingredients such as antiperspirants from pressurized, aerosol-type containers, have followed two courses. One course has been to formulate the product into a solution containing the active ingredient, whereby the formulation is in solution form within the pressure container, as discharged from the container, and as applied to a target surface. The other course has been to formulate the active ingredient into an emulsion or a dispersion of the active ingredient as a solid phase, wherein the active ingredient is in solid form within the container, and remains in solid form as the emulsion or dispersion is discharged, and upon application to a target surface.

For instance, U.S. Patent No. 3,928,545, U.S. Patent No. 3,947,556, and U.S. Patent No. 3,904,741 disclose alcohol-soluble complexes of basic aluminum chlorides with zirconyl or zinc compounds, which are said to be useful in preparing aerosol antiperspirant sprays. These patents have as their objective the formation of solutions characterized in that the antiperspirant active ingredient remains solubilized in the container and upon discharge therefrom. These patents speak of the solutions having good "fluorocarbon compatibility" which refers to the ability of the solution to retain all of the active ingredient in solution even in the presence of fluorocarbon propellants within the pressurized container, such that

the active ingredient does not precipitate from solution as a solid.

Other patents describe similarly alcoholic or hydro-alcoholic solutions of antiperspirants, and add that having the active ingredient take solid form within the pressurized container can be tolerated so long as the product that is dispensed contains the active ingredient in solid form which is also solid (powder) when the dispensed product strikes the skin.

For example, U.S. Patent No. 3,981,986 and U.S. Patent No. 3,991,176 disclose antiperspirant complexes which comprise a combination of a basic aluminum-polyol compound, a zirconium compound and an organic buffer. The complexes are said to be capable of being used in conventional antiperspirant forms, including aqueous solutions, aerosol sprays (including powder-in-oil aerosol sprays) as well as creams, lotions, and cream sticks. This patent further states that the complexes can be formed in the container and dispensed as a powder-in-oil aerosol spray wherein the antiperspirant complex is a solid which is dispersed in a non-solubilizing polar organic liquid. Thus, the antiperspirant is in solid form within the dispenser and remains in the solid form as discharged and applied to the skin.

Similarly, U.S. Patent No. 3,288,681 discloses an aerosol antiperspirant powder spray formed from a dispersion containing an aluminum antiperspirant compound, an alcohol, and a propellant. The product is formulated by selecting the compounds and the relative amounts of the compounds such that the antiperspirant is

in the form of a solid in liquid dispersion within the aerosol container and remains in insoluble form as it is discharged from the container and applied to the skin.

U.S. Patent No. 3,876,758 discloses aerosol dispensing systems for antiperspirants including an antiperspirant component which is insoluble within the container, and remains in solid form upon application to the skin.

U.S. Patent No. 3,873,686 discloses an aerosol antiperspirant formulation which, according to the patent, was converted into a powder immediately on leaving the aerosol container and landed on the human skin in the form of a powder.

Some products use a volatile liquid combined with product in a sealed container such as roll-on deodorants or antiperspirants or the like. When the product is applied from the container with a suitable dispensing system, the volatile liquid portion of the product evaporates leaving the desired active ingredient.

It can thus be seen that formulation practice with pressurized dispensing systems, especially systems for dispensing difficulty soluble active ingredients, has been to formulate the active ingredient into a composition whose form within the dispenser is the same as the form that the product is desired to exhibit following discharge. That is, products which are desired to be in liquid solution form upon application are formulated within the pressurized container in combination with propellant as solutions, and products desired to be in the solid form upon application are

formulated as a solid phase with propellant within the container.

The present inventors have realized that additional heretofore unprecedented freedom and flexibility can be realized by adopting a new course in the formulation of compositions to be dispensed from active solids in dispersion in the container to active ingredient in solution on application to the target.

The invention involves the formation of a solution of one or more active ingredients in a suitable solvent which is admixed with a volatile propellant or volatile liquid. On addition of a propellant, or volatile liquid, the propellant mixes with the solvent causing one or more of the active ingredients to precipitate in the homogeneous solvent-propellant admixture. On dispensing the dispersed, at least partially insoluble active ingredient in the propellant and solvent mixture from a pressurized container (aerosol can), the propellant quickly evaporates and the previously insoluble active ingredient redissolves to form a clear (or slightly cloudy initially) solution for application. Alternatively, a volatile liquid evaporates when a container is opened and its contents applied, leaving the desired active ingredient in soluble and activated form.

Disclosed are compositions and methods of forming and applying the composition. Particularly preferred are antiperspirant compositions solubilized in alcohol and/or water and mixed with a volatile propellant such as difluoroethane where the solvent and propellant form a homogeneous mixture of suspended

active ingredients within the pressure container but when dispensed yield an essentially clear antiperspirant solution of active ingredients without evidence of solids on the skin.

More particularly, the present invention is directed to a composition capable of being dispensed from a container comprising a admixture of: at least one active ingredient; an amount of solvent effective to dissolve said active ingredient at room temperature and atmospheric pressure; and a volatile propellant or liquid capable of forming a homogeneous mixture with said amount of solvent; at least a portion of said active ingredient being insoluble and dispersed in said homogeneous mixture, whereby on dispensing the admixture, the admixture is capable of separating into volatile propellant or liquid and a solvent containing one or more active ingredients dissolved therein.

The present invention is further directed to a method of preparing a product for dispensing from a container comprising admixing: at least one normally solid active ingredient; an amount of solvent effective to dissolve the active ingredient at room temperature and atmospheric pressure; and a volatile propellant or liquid capable of forming a homogeneous mixture with said amount of solvent at least a portion of said active ingredient being insoluble and dispersed in said homogeneous mixture; said admixture effective on being dispensed to separate into volatile propellant; and a solvent containing one or more active ingredients dissolved therein.

The Figure is a cross-sectional schematic view of a dispenser useful in the practice of the present invention.

The present invention is useful for dispensing any of a wide variety of products. Examples include products which may be applied as solutions to target surfaces, such products including for example antiperspirants, herbicides, pesticides, insect repellents, coating compositions, adhesives, and the like. Other examples include compositions which are utilized as solutions which are simply sprayed into the ambient atmosphere, without necessarily being directed to a specific target surface. Examples of the latter compositions include perfumes, room deodorizers, fumigants, disinfectants, and the like. Other products include the active ingredient in suitable solvent and a volatile liquid which evaporates when the container is opened.

Without being bound by any particular limitation as to form, the solutions upon discharge are a solution of active ingredient completely dissolved in a solvent for the active ingredient. As will be described further herein below, the solution forms in the ambient atmosphere essentially immediately upon being discharged from the container. Thus, when the composition is intended to be applied to a target surface, it is preferably already in solution form when it reaches that surface. In some embodiments, however, it is permissible that the solution form upon impact with the target surface. It should be recognized that

compositions within the scope of this invention may comprise more than one active ingredient of interest.

The dispensable composition is formed by dissolving one or more active ingredients in a suitable solvent which can comprise one or more components to form a clear solution. Solutions approaching the solubility limit of the actives in the solvent can be used to provide maximum active ingredient in the composition where needed. Lower amounts may be employed depending on the requirements of the application. Once the solution is prepared, the solution is contacted with propellant under pressure or admixed with a volatile liquid. The propellant is selected to form a homogeneous mixture of solvent and propellant although excess propellant may be employed. The volatile liquid is any material forming at atmospheric pressure or above a homogeneous admixture with the solvent wherein the volatile liquid will evaporate rapidly when the container is opened. The homogeneous mixture of solvent and propellant or volatile liquid lowers the solubility of active ingredient in the solvent causing the active ingredient(s) to precipitate from the mixture as a solid, often first appearing cloudy and then gelatinous before heavy precipitation of solids occur. The amounts of solvent and active ingredients and propellant or volatile liquid are adjusted to produce a solid dispersion of the active ingredient which is suspended or can be suspended in the homogeneous solvent and propellant or volatile liquid mixture by shaking the mixture.

On dispensing the dispersion formed previously from a container, the propellant or volatile liquid evaporates and the active ingredient re-dissolves to form a solution which may be cloudy for an instant as the solids re-dissolve in the solvent. Almost immediately after evaporation of the propellant or volatile liquid, the active ingredient is totally dissolved in the solvent providing a solution which forms a clear film on the target. Thus a solid dispersion of "Chlorhydrol" or other active ingredient in solvent and propellant under pressure can be applied to the body or other target as a clear solution already in effective form without unsightly powder present. On dispersing the packaged dispersion of active ingredient in solvent and volatile liquid, the volatile liquid evaporates and the active ingredient re-dissolves in the solvent during or shortly after application.

Any homogeneous solvent and propellant mixture can be employed. We have found as propellants non-chlorinated fluorocarbons, low molecular weight ethers, hydrocarbons, such as lower alkanes, either alone or mixed with each other or mixed with other propellants produces excellent results allowing the loading of high levels of active ingredient in the mixture of solvent and propellant under pressure but providing an essentially clear propellant free solution of active ingredient for application at room temperature and pressure.

The mixture can be prepared under pressure to form the dispersion of active ingredient in a solvent and propellant homogeneous mixture so long as the amount

of active ingredient is maintained below the solubility maximum of the active ingredient in the solvent at the application conditions of temperature and atmospheric pressure. The solution can be conveniently filled at atmospheric conditions into aerosol cans in a conventional manner. The clear solutions of active ingredient in solvent can be filled with propellant.

One aspect of the present invention comprises a system and method for dispensing an active ingredient, the system comprising a sealed, valved container which holds a gaseous propellant, the propellant comprising one or more gas components, the container further holding a dispersion of solid active ingredient slurried in a solution of one or more of said gas components dissolved in a liquid solvent for said active ingredient, the amount of said active ingredient in said dispersion not exceeding the solubility limit thereof in said solvent, wherein the container is fitted with externally actuatable valve means for dispensing said dispersion from said container under pressure exerted by said propellant wherein upon dispensing of said dispersion said one or more dissolved gas components are liberated therefrom and the dispensed solvent completely dissolves the dispensed active ingredient. The method is to establish a dispersion of a solid active ingredient slurried in a solution of one or more of said gas components dissolved in a liquid solvent for said active ingredient, wherein the active ingredient is present in said dispersion in an amount which does not exceed the solubility limit thereof in said solvent, and dispensing said dispersion from said container whereupon

said one or more dissolved gas components evaporates from the mixture allowing the dispensed solvent to redissolve the active ingredient.

With reference to the Figure, dispensing system 1 includes a container 2. Container 2 is a sealed enclosure to which is fitted externally actuatable dispensing means 3 for dispensing product when desired from within container 2 to the ambient atmosphere or to a target surface. Within container 2 are a dispersion 4 of active solids in a homogeneous mixture of solvent and propellant 5.

Container 2 is preferably formed of metal, or rigid plastic, which is inert to the contents of the container. The container can be formed of one integral piece, such as a drawn aluminum can, or it can be formed in a conventional manner from several pieces including pieces forming the sides and the upper shoulders to which dispensing means 3 is fitted and a piece forming the bottom which is fixed to the bottom edge of the side of the container throughout the circumference thereof. When desired, the interior of container 2 can be provided with a coating to protect the container material from corrosion or other adverse reactions with the contents thereof. Suitable treatment can include shellac or a thin polymeric barrier film applied to the interior of container 2. Barrier packages may also be used.

Dispensing means 3 includes a button 6, valve means 7, and dip tube 8. Valve means 7 is preferably of the type having a passage for product being dispensed, and a valve operatively coupled to a spring means which

urges the valve to a normally closed position preventing passage of product therethrough except when the valve is urged to an opening position by external actuation. Button 6 is fitted to the discharge end of the passage and contains an orifice appropriately configured to permit passage therethrough of the solid and liquid components of dispersion 4. The orifice of button 6 is also appropriately dimensioned to provide the desired spray pattern, including the desired angle through which product is dispensed i.e. as a stream or spray, and the desired droplet size, i.e., as a spray, a mist or a fog of micro droplets.

Dip tube 8 is attached to the end of valve means 7 within container 2 so as to be in fluid communication with the discharge passage. Dip tube 8 is a narrow, hollow tube dimensioned to convey the solid and liquid components of dispersion 4. Preferably, the lower end of dip tube 8 is at or near the bottom of container 2, to maximize the amount of dispersion 4 that can be discharged from the container before the contents are considered to be fully spent.

Dispersion 4 comprises an intimate slurry of a solid phase slurried in a homogeneous solvent and propellant or volatile liquid mixture. The solid phase includes the active ingredient to be dispensed. A portion of the active ingredient may also be dissolved in the solvent propellant mixture.

Active ingredients useful in the present invention include solids which form solutions, in solvents that are liquid at room temperature and at atmospheric pressure. Any such solution should

preferably contain dissolved therein a sufficient amount of the active ingredient such that the active ingredient is effective for its intended function. For instance, a solution of an antiperspirant would be desired to contain about 2% to about 10% or more by weight, of antiperspirant upon application of the solution to the skin. The amount of any other active ingredient that should be present in a solution thereof, to be dispensed from the dispensing systems of the present invention, will vary according to the identity of the active ingredient and its desired function. Such amounts can readily be determined from reference literature or from simple experimentation, for any particular active ingredient. The maximum amount of active ingredient that can be dissolved in any particular solvent or solvent system is determined by the "solubility limit", by which term is meant the maximum amount of an ingredient that can be completely dissolved in a given volume of solvent in which no propellants or volatile liquids are dissolved, at atmospheric pressure and the temperature at which the composition is to be dispensed.

Solvents useful in the dispensing systems of the present invention are generally characterized as being liquid at room temperature and pressure with varying degree of volatility and are capable of solubilizing the desired active ingredient at room temperature and pressure and are also capable of dissolving a portion of the gaseous propellant or volatile liquid employed in the dispensing systems of the present invention. Where that gaseous propellant is composed of more than one component, satisfactory

solvents include those which can solubilize all such propellant components to form a homogeneous propellant and solvent mixture.

Suitable solvents for varying active ingredients include water, mono and polyhydric alcohols of two or more carbons such as lower alkanols, such as ethanol, n-propyl alcohol, isopropyl alcohol, butanol, polyols such as glycerine and propylene glycol, dipropylene glycol, polyethylene glycols and the like; esters, including lower alkyl esters of lower alkanoic acids, such as ethyl acetate; ethers, such as diethyl ether and methylethyl ether; alkanes; hydrocarbons, kerosene, oils such as mineral or vegetable oils and lower alkyl ketones, such as acetone and methylethyl ketone. Satisfactory solvents can also include one-phase mixtures of any of the foregoing. For instance, satisfactory solvents for a system in accordance with the present invention for dispensing antiperspirant can comprise water, alcohol or a water-alcohol mixture, depending upon the identity of a particular compound or compounds used as the antiperspirant active ingredient. The amount of solvent is generally 20 to 60 wt. %, preferably 25 to 55 wt. %.

The relative amounts of active ingredient, solvent, and propellant are chosen to provide the properties within the container and upon dispensing that are desired as taught herein; identification of effective amounts of each component is a straightforward matter particularly with the guidance provided by the Examples and Tables herein.

Propellant system 5 is sealed within container 2. Sufficient propellant is present to exert a positive pressure on dispersion 4 such that when valve means 3 is actuated, an amount of dispersion 4 is forced from within container 2 to the atmosphere.

Propellants useful in the practice of the present invention can generally include any liquefiable or compressible gaseous propellant conventionally used in aerosol-type dispensers. For example, propellants are selected from the group consisting of non-chlorinated fluorocarbons, low boiling ethers, low boiling hydrocarbons and mixtures thereof. Specific examples included 1,1-difluoroethane, tetrafluoroethane, and other non-chlorinated fluorocarbons, particularly those with minor environmental consequence, propane, isobutane, n-butane, dimethyl ether, and the like.

Suitable volatile liquids would be those compounds which are liquid under conditions of use but easily volatilized or evaporated under normal atmospheric conditions or body heat or the like on application from a container.

The propellant, or volatile liquid, single compound or mixture, is selected to form a homogeneous mixture with the active ingredient laden solvent usually combined under pressure. At that point, solids dissolved in the solvent precipitate forming a dispersion of solid active ingredient in a homogeneous mixture of propellant and solvent. Some active ingredient may remain dissolved in the solvent and perhaps in the propellant, or volatile liquid, depending on solvent properties of active ingredient. Simple

tests of selected solvent and propellant or volatile liquid in glass pressure containers can be used to determine homogeneous mixtures. Two phase mixtures, while they would work with vigorous shaking prior to application, are not preferred because the active ingredient concentrates in one of the phases and may be non-uniformly dispersed when the phases are not well mixed. Excess propellant, in small quantity, may exist as a separate phase for use in pressurizing the container where necessary. The propellant solvent mixture is selected, however, to give a homogeneous mixture containing the dispersed active ingredient solids under pressure in the aerosol container, and on dispensing a solution of active ingredient and solvent as the propellant vaporizes at atmospheric pressure.

In summary, the solvent making up the liquid phase of dispersion 4 and the composition of the propellant phase 5 are selected so that a portion of the propellant is dissolved within the liquid phase of the dispersion when the contents within container 2 are fully pressurized. Distributing the propellant between the gas phase 5 and the liquid phase of dispersion 4 displaces active ingredient from solution in the solvent and assists in maintaining the desired slurry of solid active ingredient in the solvent. The active ingredient may be distributed between the solid phase of dispersion 4 and the solvent propellant forming the liquid phase of dispersion 4. On dispensing the active ingredient re-dissolves in the solvent as propellant is vaporized.

The dispensing system of the present invention can be produced using techniques conventionally employed

for manufacturing aerosol dispensing systems, with but minor modifications. A solution is prepared of the active ingredient dissolved in the solvent system (comprising a single compound or a one-phase mixture of compounds and/or solvents as the case may be). The amount of active ingredient employed should not exceed the solubility limit of that material in the solvent system employed. In that way, the amount of active ingredient present in the dispersion that subsequently forms on addition of propellant --that is, present in solution prior to addition of propellant or slurried in solid form after addition of propellant and sealing the can-- is also no greater than the solubility limit of the active ingredient in the solvent when sprayed to atmosphere or the target (propellant has evaporated).

An amount of solution containing solubilized active ingredient is charged to the container 2. This solution can be fed into the container using conventional means. The propellant of choice for the given system is fed into the container. The container can be sealed before or after adding propellant. Propellant can be fed using either through-the-valve or under-the-cup filling techniques conventionally employed in this industry for charging propellant gas to a pressurized aerosol container. As the amount of propellant present within the container increases, an equilibrium portion thereof dissolves in the liquid solvent present in the container. As the solubility of the active ingredient in the solution of the propellant and solvent decreases to become less than the solubility of the active ingredient in the solvent per se,

continued feeding of the propellant into the container and solubilization thereof in the solvent causes the active ingredient to form a precipitate which remains slurried in the solvent. The propellant should not interact with the active ingredient or the solvent as by undergoing a chemical reaction or forming an indispersible gel or other by-product. Determining propellants that satisfy these criteria is a straightforward matter. In some cases the entire amount of active ingredient will precipitate from the solvent, whereas in other cases only a portion thereof will precipitate such that the active ingredient remains distributed between the solid phase and the solvent.

Sufficient propellant is charged to the interior of the container 2 to precipitate active ingredient from solution in the solvent and to establish over the dispersion which is thus formed a sufficiently high pressure such that when the dispensing means 3 is actuated, the propellant drives the solid and solvent components of the dispersion up dip tube 8 and out through the dispensing means 3 to the atmosphere. The final pressure of the fully charged dispenser is generally about 20 to about 120 psig. The amount of propellant in the container will generally be about 10% or more by weight of all ingredients, preferably 25-90%, and more preferably about 30 to about 70% by weight.

When the dispersion is thereafter discharged from the container, the portion of the propellant gas which had been dissolved in the solvent portion of the dispersion evaporates into the atmosphere, and the solid active ingredient that was dispensed rapidly

resolubilizes into the solvent portion that had been discharged from the container. As a result, the dispensing system of the present invention effectively provides a solution of active ingredient in a solvent therefor to the atmosphere or to any particular desired target surface. This avoids the normal heavy spray of solids when dispensing a dispersion of solids from the aerosol can. If desired, preformed mixtures of active ingredient, solvent, and propellant can be prepared and the aerosol container 2 filled with the mixture under pressure. If desired, solid active ingredient can be added to solvent and propellant to form such mixtures for filling provided the active ingredient is formulated to dissolve in the solvent when propellant vaporizes from the mixture during use.

Products can also be prepared from active ingredient dissolved or combined with a solvent therefore, and a volatile liquid which can be packaged conventionally in pressure and non-pressure packages. On dispensing the product from the container, such as a liquid antiperspirant, the volatile liquid evaporates and the active ingredient re-dissolves in the solvent thereby being more available than a powder version of the same active ingredient.

Applying the active ingredient as a solution affords a number of advantages including even and thorough application; and the ability to adhere to surfaces to which solids would not readily adhere. In addition, for products whose appearance upon application is a significant factor, application in the form of a solution is preferable because the solution is clear and

leaves no unsightly residue whereas application of a powder will create a visible residue or film. Antiperspirants are a significant example of such a product whose appearance upon discharge is an important factor affecting the attractiveness and salability of the product. In addition, applying the active ingredient as a solution avoids the necessity of dissolving the active ingredient after it has been dispensed as occurs when antiperspirants (as in powder-in-oil aerosols) must be activated and solubilized by body moisture. In addition, faster drying formulas with less irritating spray particles can be developed.

Another significant advantage of the invention described herein is that it permits the dispensing of a solution containing active ingredient in higher amounts than previously achieved. The system holds more active ingredient than can be kept in solution within the pressurized container, yet manages to dispense that active ingredient in a completely solubilized form.

The invention permits tailoring the amounts of each component, particularly the propellant, to achieve desirable results unconstrained by the solubility of the active ingredient in the homogeneous solution formed by the propellant in the solvent. Increasing the amount of propellant --which is now permitted by the present invention, even as the active ingredient precipitates-- permits establishing better atomization and quicker drying of the dispensed solution. The invention also permits using less propellant per amount of active ingredient.

The present invention also permits the formulation of dispensing systems using propellants that are environmentally benign, without being unduly limited to systems that remain in solution form in the pressurized container.

EXAMPLE 1

Pressurized dispensing systems for antiperspirants were prepared containing the components listed in the following Table 1, in the amounts shown in the table. Solutions were prepared containing all the components except the propellant. The solution was sealed in valved glass bottles, and then the indicated amount of propellant was injected through the valve.

A powder was seen to precipitate upon addition of the propellant. The powder was a fine white dispersion that dispersed easily upon gentle shaking, and thereafter settled slowly. When samples of these preparations were sprayed, under pressure from the propellant, the spray did not contain noticeable solid particles and rapidly formed a solution which was clear on the skin and rapidly dried to an unnoticeable film.

TABLE 1

AMOUNTS (wt. %)

Component	Test No.:	1	2	3	4	5	6	7	8	9
Chlorhydrol (50% Aqueous Solution)		15.0	15.0	15.0	15.0	15.0	-	-	-	-
REACH 501 (50% Aqueous Solution		-	-	-	-	-	15.0	15.0	15.0	15.0
SE#40 Ethanol (200 proof)		42.8	39.8	45.0	41.8	41.8	41.8	41.8	39.8	45.0
Propylene glycol		2.0	5.0						5.0	-
Polyethylene glycol (PEG 200)		-	-	-	3.0	-	3.0	-	-	-
Isopropyl palmitate		-	-	-	-	3.0	-	3.0	-	-
Propellant (1,1-difluorethane)		40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0
Fragrance		0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	-

EXAMPLE 2

Pressurized dispensing systems for antiperspirants were prepared containing the components listed in the following Tables 2, 2A, and 2B, in the amounts shown. The solution was sealed in valved glass bottles and the amount of propellant injected through the valve. Various amounts of active ingredient indicate the advantage of forming a precipitate within the can or jar which is soluble in solvent on dispensing, in that more active ingredient can be dispensed and the amount of propellant used per unit of active ingredient can be reduced. There is some change in the amount of active ingredient which is soluble in the solvent propellant solution in the pressure container over time.

TABLE #2

Effect of Reach 501 Concentration with Various Propellants

Amount - % by weight

Example	1	2	3	4	5	6	7	8	9	10	11	12
Reach 501 (50% Aqueous Solution)	15.0	12.5	10.0	7.5	15.0	12.5	10.0	7.5	15.0	12.5	10.0	7.5
SD#40 Anhyd. (100% ethanol)	45.0	47.5	50.0	52.5	45.0	47.5	50.0	52.5	45.0	47.5	50.0	52.5
P152A 1,1-Difluorethane)	40.0	40.0	40.0	40.0	-	-	-	-	-	-	-	-
P-152A/A31 75% 1.1-Difuoroethane 25% Isobutane	-	-	-	-	-	-	-	-	40.0	40.0	40.0	40.0
DME/A31 60% Dimethyl ether 40% Isobutane	-	-	-	-	40.0	40.0	40.0	40.0	-	-	-	-
% Propellant Product Still Clear	36.0	37.2	39.7	40.0	26.4	31.8	36.7	39.5	31.0	31.8	36.6	40.0
% Propellant @ Approx. PPT. Point	37.6	37.8	40.0	-	28.2	34.9	37.8	-	32.0	32.8	37.9	-
Finish Product All Propellant Added Day 1 @ R.T.	Powder Susp.	Milky Gel	Milky	Hazy Soln.	Powder Susp.	Powder Susp.	Milky	Milky Sl.Gel	Powder Susp.	Powder Susp.	Milky	Hazy Soln.
Day 10 @ R.T.	Powder Susp.	Gels Thins @ Shake	Gels Thins @ Shake	Milky Soln.	Powder Susp.	Powder Susp.	Milky @ Gel Thins	Gels Thins @ Shake	Powder Susp.	Powder Susp.	Powder Sl. Gel Thins @ Shake	Gels Thins @ Shake

TABLE 2A

EFFECT OF VARYING CHLORHYDROL AND PROPELLANT CONTENTS
(all amounts in wt.%)

Test No.	1	2	3	4	5	6	7	8
Chlorhydrol (50% Aq. Solution)	15.0	12.5	10.0	7.5	20.0	17.5	15.0	12.5
SD # 40 (100% ethanol)	45.0	47.5	50.0	52.5	40.0	42.5	45.0	37.5
P152A (1,1-difluoroethane)	40.0	40.0	40.0	40.0	40.0	40.0	40.0	50.0
Max.% Propellant w/ Product Still Clear	25	34	40	40	---	---	---	---
% Propellant at Start of Precip.	18.5	36.2	---	---	28.8	32.5	---	---
Final Product Appearance Held at Room Temperature at : day 1	Powder susp.	Milky Starts to gel	Hazy No ppt.	Clear	Powder susp.	Powder susp.	Powder susp.	Powder susp.
day 3	---	---	---	---	Powder susp.	Powder susp.	---	---
day 5	---	---	---	---	---	---	Powder susp.	Powder susp.
day 10	Powder susp.	Gel	Gel thins with shaking	Slight hazy solut.	---	---	---	---

TABLE 2A - CONTINUED

EFFECT OF VARYING CHLORHYDROL AND PROPELLANT CONTENTS
(all amounts in wt.%)

Test No.	9	10	11	12	13	14	15	16
Chlorhydrol (50% Aq. Solution)	10.0	5.0	2.5	17.5	14.6	11.7	5.8	2.9
SD # 40 (100% ethanol)	30.0	15.0	7.5	42.5	35.4	28.3	14.2	7.1
P152A (1,1-difluoroethane)	60.0	80.0	90.0	40.0	50.0	60.0	80.0	90.0
Max.% Propellant w/ Product Still Clear	---	---	---	---	---	---	---	---
% Propellant at Start of Precip.	---	---	---	---	---	---	---	---
Final Product Appearance Held at Room Temperature at : day 1	Powder susp.	Powder susp.	Powder susp.	Powder susp.	Powder susp.	Powder susp.	Powder caked to bottom	Powder caked to bottom
day 3	---	---	---	---	---	---	---	---
day 5	Powder susp.	Powder is susp. and caked on bottom	Powder is susp. and caked on bottom	Powder susp.	Powder susp.	Powder susp.	same as above	same as above
day 10	---	---	---	---	---	---	---	---

TABLE 2B

EFFECT OF VARYING CHLORHYDROL CONTENTS
(all amounts in wt. %)

Test No.	1	2	3	4	5	6	7	8	9	10	11	12
chlorhydrol (50% Aq. Solution)	15.0	12.5	10.0	7.5	17.5	20.5	15.0	12.5	10.0	7.5	20.0	17.5
SD # 40 Ethanol	45.0	47.5	50.0	52.5	42.5	40.0	45.0	47.5	50.0	52.5	40.0	42.5
P152A/A31: 75%-1, 1-difluoroethane 25%-isobutane	40.0	40.0	40.0	40.0	40.0	40.0	---	---	---	---	---	---
DME/A31: 60%-dimethylether 40%-isobutane	---	---	---	---	---	---	40.0	40.0	40.0	40.0	40.0	40.0
Max.% Propellant w/ Product Still Clear	28.0	30.0	33.3	40.0	---	---	28.4	31.5	31.9	40.0	---	---
% Propellant at Start of Precip.	31.2	31.5	34.6	---	---	---	29.4	33.0	35.3	---	---	---
Final Product Appearance Held at Room Temperature at: day 1	Powder susp.	Powder susp.	Powder susp.	Hazy solu.	Powder susp.	Large particles settling	Powder susp.	Powder, slight gel	Milky, starts to gel	Hazy starts to gel	Powder susp.	Powder susp.
day 3	---	---	---	---	---	---	---	---	---	---	Powder susp.	Powder susp.
day 7	---	---	---	---	Powder susp.	Galled to bottom	---	---	---	---	---	---
day 10	Powder susp.	Powder susp.	Powder susp.	Milky Powder	---	---	Powder susp.	Powder with gel, thins with shaking	Powder with gel, thins with shaking	Gel, thins with shaking	---	---

WHAT IS CLAIMED IS:

1. A composition capable of being dispensed from a container comprising a admixture of:

at least one active ingredient;

an amount of solvent effective to dissolve said active ingredient at room temperature and atmospheric pressure; and

a volatile propellant or liquid capable of forming a homogeneous mixture with said amount of solvent;

at least a portion of said active ingredient being insoluble and dispersed in said homogeneous mixture, whereby on dispensing the admixture, the admixture is capable of separating into volatile propellant or liquid and a solvent containing one or more active ingredients dissolved therein.

2. A composition according to Claim 1 wherein said active ingredient is an antiperspirant.

3. A composition according to Claims 1 or 2 wherein the solvent is selected from the group consisting of water, lower alcohols, glycols, esters, ethers, ketones and mixture thereof.

4. A composition according to any of Claims 1-3 wherein the propellant is selected from the group consisting of non-chlorinated fluorocarbons, low boiling ethers, low boiling alkanes, and mixtures thereof.

5. A composition according to any of Claims 1-4 wherein the propellant is selected from the group consisting of normal butane, propane, difluoroethane, tetrafluoroethane, isobutane, dimethyl ether and mixtures thereof.

6. A composition according to any of Claims 1-5 wherein said propellant comprises at least about 10% or more of the homogeneous mixture.

7. A composition according to any of Claims 1-6 where the propellant is selected from the group consisting of non-chlorinated fluorocarbons, low boiling ethers, low boiling alkanes, and mixtures thereof.

8. The composition according to any of Claims 1-7 wherein the solvent is selected from the group consisting of water, lower alcohols, polyols, esters, ethers, ketones and mixtures thereof.

9. A method of preparing a product for dispensing from a container comprising admixing:

at least one normally solid active ingredient;

an amount of solvent effective to dissolve the active ingredient at room temperature and atmospheric pressure;

and a volatile propellant or liquid capable of forming a homogeneous mixture with said amount of solvent at least a portion of said active ingredient being insoluble and dispersed in said homogeneous mixture;

said admixture effective on being dispensed to separate into volatile propellant;

and a solvent containing one or more active ingredients dissolved therein.

10. The method of Claim 9 wherein said active ingredient is an antiperspirant.

11. The method of Claims 9 or 10 wherein said solvent is selected from the group consisting of water,

lower alcohols, glycols, esters, ethers, ketones and mixtures thereof.

12. The method of any of Claims 9-11 wherein the propellant is selected from the group consisting of non-chlorinated fluorocarbons, low boiling ethers, low boiling alkanes, and mixtures thereof.

13. The method of any of Claims 9-12 wherein the propellant is selected from the group consisting of difluoroethane, tetrafluoroethane, isobutane, normal butane, propane, dimethyl ether and mixtures thereof.

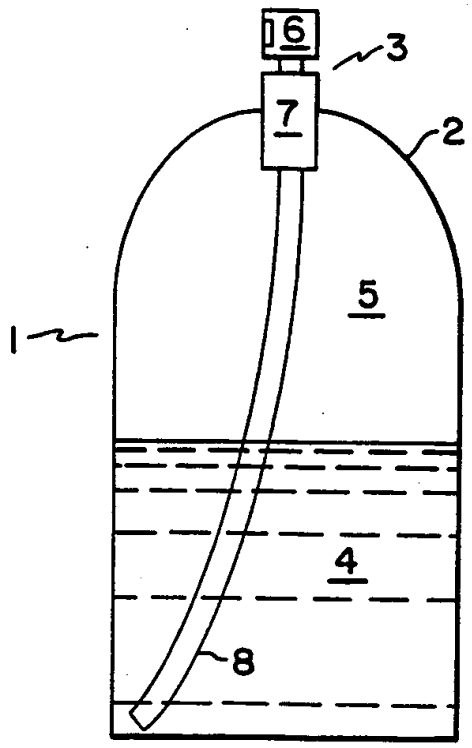
14. A system for dispensing an active ingredient, the system comprising a sealed, valved container and within the container a gaseous propellant, the propellant comprising one or more gas components, the container further holding a dispersion comprising solid active ingredient slurried in a solution of one or more of said gas components dissolved in a liquid solvent for said active ingredient, the amount of said active ingredient in said dispersion not exceeding the solubility limit thereof in said solvent, and externally actuatable valve means fitted to said container for dispensing said dispersion from said container under pressure exerted by said propellant wherein upon dispensing of said dispersion said one or more dissolved gas components are liberated therefrom and the dispensed solvent completely redissolves the dispensed active ingredient.

15. A system according to Claim 14 wherein said solvent is selected from the group consisting of water, lower alcohols, glycols, esters, ethers, ketones and mixtures thereof.

16. A system according to Claims 14 or 15 wherein said propellant is selected from the group consisting of non-chlorinated fluorocarbons, low boiling ethers, low boiling alkanes, and mixtures thereof.

17. A system according to any of Claims 14-16 wherein said active ingredient is an antiperspirant.

Figure



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/07513

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) :A61K 9/08; 7/32
US CL :424/401

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 424/401, 46, 65, DIG 1; 252/305; 222/402.1

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 5,118,494 (SCHULTZ ET AL) 02 June 1992; see entire document.	1-3, 9-11, 14-16
Y	Research & Development, "Fluorocarbon and Dimethyl Ether Aerosol Propellants", (STERLING), December 1982, pages 50-52.	1-3, 9-11, 14-16
Y	US, A, 3,981,986 (RUBINO) 21 September 1976; see entire document.	1-3, 9-11, 14-16

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

*	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be part of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E"	earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search	Date of mailing of the international search report
25 AUGUST 1994	08 NOV 1994

Name and mailing address of the ISA/US	Authorized officer
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Form PCT/ISA/210 (second sheet)(July 1992)*

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/07513

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☒ Claims Nos.: 4-8, 12, 13 & 17
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.